Synthesis of large wadsleyite single crystals by solid-state recrystallization

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ABSTRACT

Single crystals of (Mg0.89Fe0.11)2SiO4 wadsleyite with dimensions up to ~1 mm were synthesized by solid-state recrystallization under high pressure. Synthesis experiments of the wadsleyite single crystals were performed at 16 GPa and 1870 K for 1–3 h using a Kawai-type multi-anvil apparatus. The wadsleyite crystals are virtually free of inclusions and cracks. Their chemical compositions are homogeneous with Fe/(Mg + Fe) of 0.112(2). Unpolarized infrared spectra indicate that the synthesized sample contains 0.15–0.30 wt% H2O. The method of synthesizing large, high-quality single crystals of wadsleyite will facilitate future measurements of physical properties including elasticity and elastic anisotropy, electrical and thermal conductivities, atomic diffusivity, and creep strength, which will improve models of the composition and dynamics of the mantle transition zone.

Keywords: Wadsleyite, single crystal, mantle transition zone, high-pressure synthesis, Kawai-type multi-anvil apparatus

INTRODUCTION

Wadsleyite is expected to be the most dominant mineral at the upper part of the mantle transition zone (MTZ) (e.g., Irifune and Isshiki 1998), and thereby it controls the physical and chemical properties of this region. Seismic observations have revealed anisotropic seismic wave propagation within the upper MTZ (e.g., Visser et al. 2008). Global seismic anisotropy in the upper MTZ has been attributed to a crystallographic preferred orientation (CPO) of wadsleyite (e.g., Kawazoe et al. 2013). However, a quantitative evaluation of the seismic anisotropy observed in the upper MTZ is difficult because the elastic stiffness constants of wadsleyite have not been experimentally determined under simultaneous high pressure and high temperature (e.g., Wang et al. 2014). Moreover, the wadsleyite CPO in the upper MTZ may cause anisotropy in other physical properties such as viscosity; cf. deformed olivine in the upper mantle (Hansen et al. 2012). Consequently, a detailed understanding of the anisotropy of the physical properties of wadsleyite is fundamental to model anisotropy of physical and chemical properties in the upper MTZ.

Many types of experiments to determine anisotropy in physical and chemical properties of wadsleyite require single crystals of sufficient size and quality. In experiments with diamond-anvil cells (DAC), single crystals larger than 100 μm are useful for the preparation of single-crystal samples with defined dimensions, shape, and, if needed, crystallographic orientation by the focused ion beam (FIB) technique (Marquardt and Marquardt 2012). In the case of multi-anvil apparatuses, atomic diffusion and deformation experiments can be performed on wadsleyite samples as small as 0.4–0.5 mm (Kawazoe et al. 2010; Shimojuku et al. 2004). Therefore, wadsleyite single crystals larger than ~0.4 mm are ideal for a range of experiments to determine the anisotropy of its physical properties under high pressure and temperature.

Previously, large wadsleyite single crystals were synthesized by solid-state recrystallization using a Kawai-type multi-anvil apparatus (Sawamoto 1986). In these experiments, wadsleyite single crystals with dimensions up to 0.5 mm were obtained near the wadsleyite–ringwoodite phase boundary (19–21.5 GPa and 1940–2670 K). However, the wadsleyite crystals contained inclusions and showed variation in Mg/(Mg + Fe) from crystal to crystal (Sawamoto 1986). We note that in this previous study, temperature was overestimated because it has been shown that MgSiO3 ringwoodite is unstable above ~2170 K (e.g., Fei et al. 2004). In another work, the temperature-gradient method was applied to single-crystal growth of wadsleyite in carbonate solutions using a Kawai-type apparatus (Shatskiy et al. 2009). In these experiments, wadsleyite single crystals with dimensions up to 1.0 mm were obtained in coexistence with quenched melt. However, the carbonate flux method produced crystals with inclusions of the solvent (Shatskiy et al. 2009).

In the present study, we synthesized high-quality single crystals of (Mg0.89Fe0.11)2SiO4 wadsleyite with dimensions up to ~1 mm by solid-state recrystallization. We describe the method and discuss sample characterization including evaluation of inclusions, cracks, chemical compositions, and water content. In addition, we outline potential applications for the synthesized crystals to study the intrinsic anisotropy of wadsleyte to many physical and chemical properties.

EXPERIMENTAL METHODS

Synthesis experiments

The starting material was a powder of San Carlos olivine [(Mg0.9Fe0.1)2SiO4]. Olivine single crystals with no visible inclusion were hand-picked under a stereomicroscope and ground to a fine powder using a mortar. The starting powder was packed in a Re foil capsule (1.6 mm outer diameter and 2.7 mm length). Neither solvent nor water was added to the starting material.

The high-pressure synthesis was performed using a 1000-ton Kawai-type multi-anvil apparatus with split-sphere type guide blocks (Keppeler and Frost 2005). The second-stage anvils with an 8 mm truncation were made of tungsten carbide (ha-7%, hawedia). The capsule was loaded into a ceramic octahedron with a 14 mm